

PCT

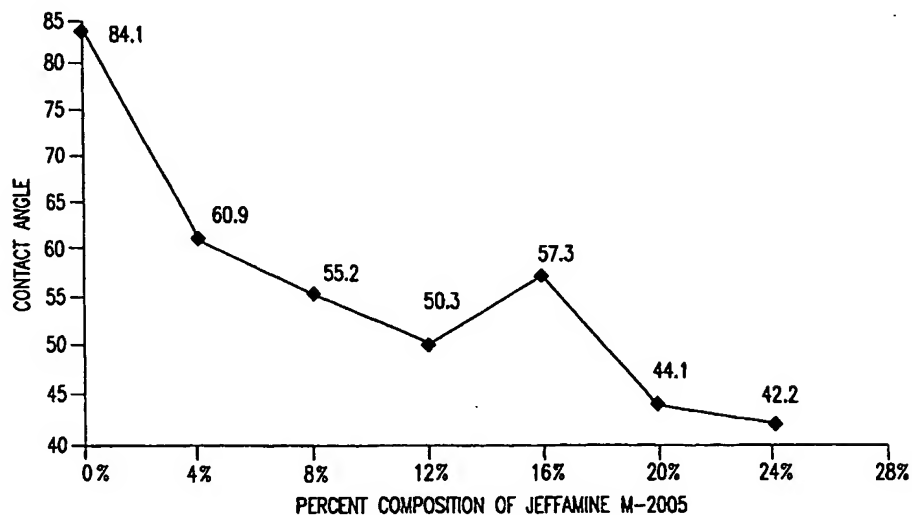
WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁷ : D04H 1/42, D01F 6/30, A61L 15/00	A1	(11) International Publication Number: WO 00/12801 (43) International Publication Date: 9 March 2000 (09.03.00)
(21) International Application Number: PCT/US99/18030 (22) International Filing Date: 9 August 1999 (09.08.99) (30) Priority Data: 09/144,501 31 August 1998 (31.08.98) US (71) Applicant: KIMBERLY-CLARK WORLDWIDE, INC. [US/US]; 401 North Lake Street, Neenah, WI 54957 (US). (72) Inventors: NING, Xin; 12431 Crabapple Meadow Way, Alpharetta, GA 30004 (US). WANG, James, Hongxue; 1325 East Overland Road, Appleton, WI 54911 (US). (74) Agents: PETERSEN, Maxwell, J.; Pauley Petersen Kinne & Fejer, Suite 365, 2800 West Higgins Road, Hoffman Estates, IL 60195 (US) et al.		(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>With amended claims and statement.</i>

(54) Title: **NONWOVEN POLYOLEFIN FABRICS HAVING HYDROPHILICITY**



(57) Abstract

A nonwoven web having durable hydrophilic properties is prepared from an initially hydrophobic polymer chemically reacted with a polar material to form a polar-modified polymer, and further chemically reacted with a hydrophilic material to impart the hydrophilic properties. The hydrophilic polymer reaction product thus prepared can be formed into a nonwoven web using conventional melt spinning techniques. Alternatively, a nonwoven web can be formed from the hydrophobic polymer or its polar-modified intermediate, and the remaining reaction or reactions accomplished by surface grafting.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LJ	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

NONWOVEN POLYOLEFIN FABRICS HAVING ENHANCED HYDROPHILICITY

FIELD OF THE INVENTION

This invention relates to chemically modified polyolefin-based nonwoven fabrics having enhanced hydrophilic properties.

BACKGROUND OF THE INVENTION

Maleated polyolefins (including, for instance, maleated polypropylene and maleated polyethylene) are typically hydrophobic. These resins are commonly used as compatibilizers and/or adhesives between polar materials, such as nylon or ethylene vinyl alcohol, and polyolefins. Typically, an anhydride such as maleic anhydride is chemically reacted (i.e., grafted) onto the polyolefin backbone chain using heat and/or a catalyst. When exposed to a polar material in the presence of heat, the grafted polyolefin forms a chemical linkage to the polar material resulting in bonding and compatibilization.

Some uses for maleated polyolefins are disclosed in U.S. Patent 5,721,315, issued to Evans et al. These uses include engineering plastics which are materials for structural members in the fields of transport machines (automobiles, ships and the like), tools, appliances, sporting goods, leisure goods, connectors, and tubes.

One use of polyolefins is in the manufacture of nonwoven fibrous webs for various applications. Nonwoven webs can include spunbond webs, meltblown webs, and bonded carded webs, for instance, and laminates of them. These webs are used in a wide variety of absorbent materials and apparel including diapers, tampons, medical garments, surgical gloves, caps, aprons, and sterilization wraps. When used in absorbent materials, the nonwoven webs may form part of the topsheet, backing or similar structural material and a breathable film laminated to the web may provide liquid barrier and moisture vapor transmission. When used in medical apparel, specific laminates of nonwoven webs may provide structural integrity and breathability as well as barrier to liquids, bacteria and viruses.

Polyolefins used to make nonwoven webs are typically hydrophobic. When a nonwoven web is intended to transmit or channel liquid, such as in a topsheet of an absorbent structure, the hydrophobic nature of the material may act as a hindrance. Various surface treatments of nonwoven webs are known for improving their hydrophilicity, rendering them more wettable to aqueous liquids. These surface treatments have certain

disadvantages, including a potential to leave the nonwoven web and escape to the wearer's skin or the inner core of the absorbent article. There is a need or desire for a polyolefin-based nonwoven web having hydrophilic properties which are more permanent, and which does not require the use of mobile surfactants.

SUMMARY OF THE INVENTION

The present invention is directed to a fibrous nonwoven web having a chemically imposed hydrophilic surface. "Chemically imposed" means that the hydrophilic surface is formed by chemical reaction and linkage between a hydrophilic moiety and an initially hydrophobic nonwoven fabric-forming material. The chemical reaction and linkage of the hydrophilic moiety is distinguishable from prior art methods in which a nonwoven fabric is rendered hydrophilic by surface coating of a hydrophilic compound, or by merely blending (and not reacting) a hydrophilic compound with a nonwoven fabric-forming polymer. The chemical reaction and linkage of the hydrophilic moiety to the nonwoven fabric material causes hydrophilic properties which are more permanent, and less transitory, than would occur without the chemical reaction.

The starting material for the invention is a hydrophobic fibrous nonwoven web, or a hydrophobic nonwoven web-forming polymer material. The nonwoven web, or the web-forming material, is chemically reacted with an anhydride and/or its carboxylic acid derivative to form an intermediate hydrophobic material having a polar functionality. The intermediate material is then further reacted with a hydrophilic compound having a reactive moiety, such as a hydroxyl or amino group, that forms a chemical linkage with the polar functionality.

The resulting product is either a hydrophilic fibrous nonwoven web, or a hydrophilic polymer material that can be spun into a fibrous nonwoven web. The web or web-forming material possesses all the desirable properties of the underlying polymeric base material, except for the hydrophilic addition. The chemically imposed hydrophilicity is durable, meaning that it cannot be washed off or otherwise physically removed.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a plot showing the effect of different reaction-inducing levels of an anhydride and a polyglycol on the water contact angle of a polyolefin. Lower water contact

angles, measured by ASTM D-5946-96, indicate a more hydrophilic material. In Fig. 1, the polyglycol level is varied at three fixed levels of maleic anhydride.

Fig. 2 is another plot showing the effect of different reaction-inducing levels of an anhydride and a polyglycol on the water contact angle of a polyolefin. In Fig. 2, the maleic anhydride level is varied for three fixed levels of polyglycol.

Fig. 3 is a plot showing the effect of different reaction-inducing levels of a polyglycol having three different weight average molecular weights, at a constant level of maleic anhydride, on the water contact angle of a polyolefin.

Fig. 4 is a plot showing the effect of different reaction-inducing levels of a polyglycol, on the water contact angle of two polypropylene materials reacted with different levels of maleic anhydride.

Fig. 5 is a bar graph showing the water contact angles of polyolefins reacted using three different levels of a polyglycol, and three different levels of maleic anhydride, before and after washing with distilled water.

Fig. 6 is a plot showing the reaction reproducibility as reflected in water contact angles for a maleated polyolefin further reacted with different levels of a polyglycol.

Fig. 7 and 8 are plots showing the water contact angles achieved after reaction of different levels of two polyglycol materials with the same maleated polypropylene.

DEFINITIONS

"Nonwoven web" means a web having a structure of individual fibers or threads which are interlaid, but not in an identifiable, repeating manner. Nonwoven webs have been, in the past, formed by a variety of processes such as, for example, melt-blowing processes, spunbonding processes and bonded carded web processes.

"Meltblown fibers" means fibers formed by extruding a molten thermoplastic material through a plurality of fine, usually circular, die capillaries as molten threads or filaments into a high velocity gas (e.g., air) stream which attenuates the filaments of molten thermoplastic material to reduce their diameter, possibly to microfiber diameter. Thereafter, the meltblown fibers are carried by the high velocity gas stream and are deposited on a collecting surface to form a web of randomly disbursed meltblown fibers. Such a process

is disclosed, for example, in U.S. Patent 3,849,241 to Butin, the disclosure of which is hereby incorporated by reference.

"Microfibers" means small diameters fibers having an average diameter not greater than about 100 microns, for example, having an average diameter of from about 0.5 microns to about 50 microns, or more particularly, an average diameter of from about 4 microns to about 40 microns.

"Spunbond fibers" refers to small diameter fibers which are formed by extruding a molten thermoplastic material as filaments from a plurality of fine, usually circular, capillaries of a spinnerette with the diameter of the extruded filaments then being rapidly reduced as by, for example, eductive drawing or other well-known spunbonding mechanisms. The production of spunbonded nonwoven webs is illustrated in patents such as, for example, in U.S. Patent 3,802,817 to Matsuki et al. and U.S. Patent 5,382,400 to Pike et al. The disclosures of these patents are hereby incorporated by reference.

"Polymer" generally includes, but is not limited to, homopolymers, copolymers, such as, for example, block, graft, random and alternating copolymers, terpolymers, etc. and blends and modifications thereof. Furthermore, the term "polymer" shall include all possible geometrical configurations of the material. These configurations include, but are not limited to, isotactic, syndiotactic and random symmetries.

"Bicomponent fibers" refers to fibers which have been formed from at least two polymers extruded from separate extruders but spun together to form one fiber. The polymers are arranged in substantially constantly positioned distinct zones across the cross-section of the bicomponent fibers and extend continuously along the length of the bicomponent fibers. The configuration of such a bicomponent fiber may be, for example, a sheath/core arrangement wherein one polymer is surrounded by another or may be a side-by-side arrangement or an "islands-in-the-sea" arrangement. Bicomponent fibers are taught in U.S. Patent 5,108,820 to Kaneko et al., U.S. Patent 5,336,552 to Strack et al., and European Patent 0586924. For two component fibers, the polymers may be present in ratios of 75/25, 50/50, 25/75 or any other desired ratios.

"Biconstituent fibers" refers to fibers which have been formed from at least two polymers extruded from the same extruder as a blend. The term "blend" is defined

below. Biconstituent fibers do not have the various polymer components arranged in relatively constantly positioned distinct zones across the cross-sectional area of the fiber. The various polymers are usually not continuous along the entire length of the fiber, but are instead in the form of fibrils which start and end at random. Biconstituent fibers are sometimes also referred to as multiconstituent fibers. Fibers of this general type are discussed in, for example, U.S. Patent 5,108,827 to Gessner. Bicomponent and biconstituent fibers are also discussed in the textbook *Polymer Blends and Composites* by John A. Manson and Leslie H. Sperling, copyright 1976 by Plenum Press, a division of Plenum Publishing Corporation of New York, ISBN 0-306-30831-2, at pages 273 through 277.

"Blend" means a mixture of two or more polymers while the term "alloy" means a sub-class of blends wherein the components are immiscible but have been compatibilized. "Miscibility" and "immiscibility" are defined as blends having negative and positive values, respectively, for the free energy of mixing. Further, "compatibilization" is defined as the process of modifying the interfacial properties of an immiscible polymer blend in order to make an alloy.

"Hydrophilic" refers to a surface or material that has an affinity for water, and is wettable by water. Some hydrophilic materials are capable of absorbing water, dissolving in water, and/or swelling. A hydrophilic material should have a water contact angle of about 80 degrees or less, measured by ASTM D5946-96.

"Hydrophobic" refers to a surface or material that is poorly wetted by water, has little or no affinity for water, and tends to repel water. A hydrophobic material may have a water contact angle of at least 80 degrees, sometimes 90 degrees or more.

"Chemically imposed hydrophilic surface" refers to a hydrophilic surface formed by chemical reaction between a hydrophilic moiety and an initially hydrophobic nonwoven web or web-forming polymer. Chemically imposed hydrophilic surfaces are generally durable, meaning that the surfaces remain hydrophilic after washing with distilled water.

"Consisting essentially of" does not exclude the presence of additional materials which do not significantly affect the desired characteristics of a given composition or product. Examples of such materials include, without limitations, pigments, antioxidants,

stabilizers, surfactants, waxes, flow promoters, particulates and materials added to enhance processability of the composition.

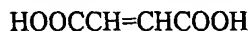
DETAILED DESCRIPTION OF THE PRESENTLY PREFERRED EMBODIMENTS

The starting material for the invention is a fibrous nonwoven web or web-forming material which is hydrophobic. The starting material includes a hydrophobic polymer. Exemplary hydrophobic polymers include without limitation, polypropylene, polyethylene (high and low density), ethylene copolymers with C₃-C₂₀ α -olefins, propylene copolymers with ethylene or C₄-C₂₀ α -olefins, butene copolymers with ethylene, propylene, or C₅-C₂₀ α -olefins, polyvinyl chloride, polyesters, polyfluorocarbons, hydrophobic polyurethane, polystyrene, acrylic resins, and combinations thereof. Polyolefins are preferred, including polyethylenes, polypropylenes, copolymers thereof, and blends thereof.

The nonwoven web may be any type of thermoplastic nonwoven web. For instance, the web may be a spunbonded web, a meltblown web, a bonded carded web, or a combination including any of the foregoing. The nonwoven web may also be a bicomponent or biconstituent web, as well as a web containing one or more of the above-listed thermoplastic polymers. In the case of a bicomponent web, for instance, it is important only that the surface material include a hydrophobic polymer which can be modified in accordance with the present invention to render it hydrophilic. The composition of a second (inner) material, not exposed at the fiber surfaces, is immaterial for purposes of the invention and may be hydrophilic or hydrophobic. The nonwoven web may have a basis weight of about 0.1-150 grams per square meter (gsm), preferably about 1-100 gsm, more preferably about 5-50 gsm.

The fibrous nonwoven web or web-forming material is chemically reacted with a polar material. The polar material can include an anhydride or anhydride derivative (e.g., a carboxylic acid derivative) and can be a monomer, polymer, or compound. The reaction product is a hydrophobic polymer material having a polar functionality (herein called a polar-modified polymer). Preferably, the nonwoven web is reacted with maleic anhydride or one of its derivatives, such as maleic acid or fumaric acid. Other suitable polar

materials include without limitation various anhydrides and their derivatives, particularly those having an unsaturated carbon-carbon double bond:



The polar material is reacted with the hydrophobic polymer, either using heat or a catalyst (e.g., a peroxide catalyst), or a combination of heat and catalyst. When heat is employed, the reaction may take place at a temperature near or above the melting point of the hydrophobic polymer. For instance, the hydrophobic polymer and polar material may be blended together in a mixer, with the hydrophobic polymer in the molten state, to facilitate substantially homogeneous mixing and reaction between the polar material and hydrophobic polymer. When the hydrophobic polymer includes polypropylene, for instance, the reaction may occur in a mixer at a temperature of about 160-225°C, preferably 175-200°C, with or without a peroxide catalyst, whereby the polar material is graft polymerized onto the hydrophobic polymer. Alternatively, the chemical reaction may occur at a much lower temperature in a solvent, with the grafting reaction being aided by a peroxide catalyst.

Techniques for graft polymerizing a polar material, such as maleic anhydride or a dicarboxylic acid derivative, onto a hydrophobic polymer (e.g., a polyolefin) are well known in the art, and do not constitute part of this invention. As an alternative to polymerizing the polar material with the hydrophobic polymer, a suitable polar-modified hydrophobic polymer may be purchased commercially. Commercially available polar-modified hydrophobic polymers include without limitation the following:

EXXELOR® 1015, a maleated polypropylene available from Exxon Chemical Co., having a melt flow rate (230°C) of 120 grams/10 minutes and containing 0.4% by weight grafted maleic anhydride;

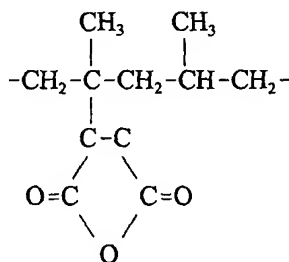
POLYBOND® 3150, a maleated polypropylene available from Uniroyal Chemical Co., having a melt flow rate (230°C) of 50 grams/10 minutes and containing 0.7% by weight grafted maleic anhydride; and

POLYBOND® 3200, a maleated polypropylene available from Uniroyal Chemical Co., having a melt flow rate (230°C)

of 110 grams/10 minutes and containing 1.0% by weight grafted maleic anhydride.

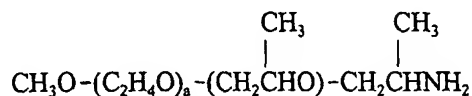
The maleated polyolefin (or other polar-modified polymer) may itself be hydrophobic and not wettable to water, or borderline between hydrophobic and hydrophilic. The reaction with the polar material does not render the polymer backbone hydrophilic; rather, it provides a chemical linkage for the subsequent reaction with a hydrophilic material. Generally, the polar-modified polymer should contain about 0.1-3.0% by weight of the polar monomer, preferably about 0.4-1.0% by weight, more preferably about 0.6-0.8% by weight.

Preferably, the polar material is grafted onto the hydrophobic polyolefin, resulting in a stereochemistry most favorable for further reaction. Maleated polypropylene, for instance, has the following stereochemistry in which the functional anhydride group projects outward from the backbone chain:



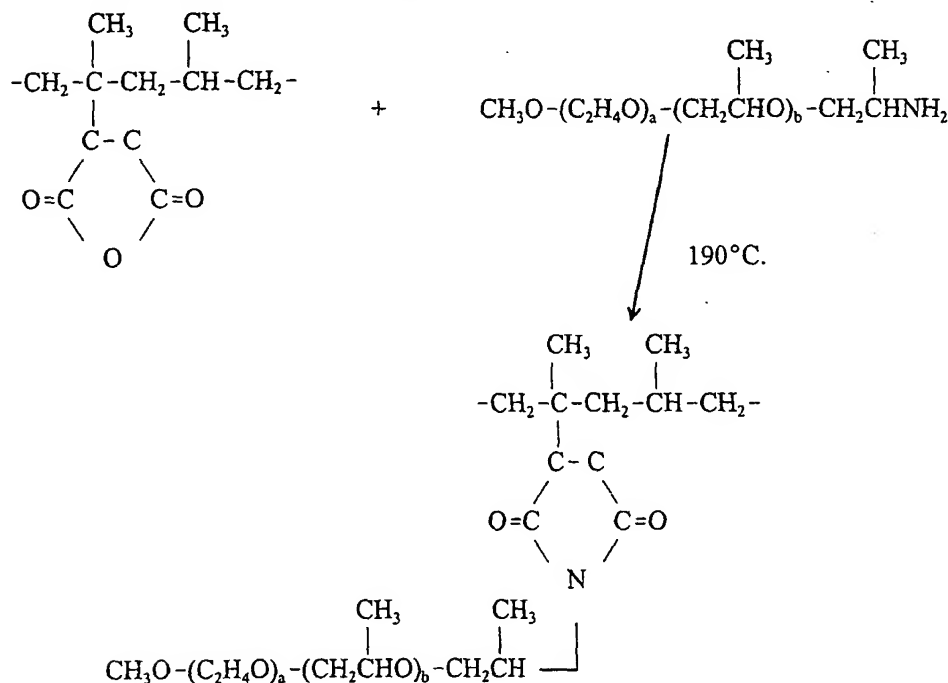
In accordance with the invention, the polar-modified hydrophobic polymer is reacted with a hydrophilic material, thereby increasing the hydrophilicity of the polymer to render it wettable to water. The hydrophilic material can be a hydrophilic monomer, polymer, compound, or blend containing one or more of these. Suitable hydrophilic materials include organic alcohols, dialcohols, tertiary alcohols, polymers containing them, and other hydrophilic materials having groups which react with the polar group (e.g., the anhydride moiety) on a polar-modified hydrophobic polymer. Presently preferred hydrophilic materials include polyglycols and polyoxides, including polyolefin glycols and oxides, such as polyethylene glycol, polyethylene oxide, polypropylene glycol, polypropylene oxide, and copolymers and mixtures thereof. Presently preferred polyglycols include those having monoamine and/or diamine linkages which further promote hydrophilicity. The JEFFAMINE® series of polyglycols, available from Huntsman Chemical

Co., includes monoamines and diamines of varying molecular weights. A typical JEFFAMINE® monoamine structure is as follows:



wherein a and b are integers.

Maleated polyolefins can be reacted with polyglycols in the presence of heat to form imides having increased hydrophilic properties:



The reaction between a polar-modified hydrophobic polymer and a hydrophilic material can be accomplished by melt blending the ingredients together, with or without a peroxide catalyst to form a hydrophilic polymer reaction product. The reaction mixture should contain about 1-35% by weight of the hydrophilic material, preferably about 4-25% by weight, more preferably about 8-20% by weight. The reaction preferably occurs with the polar-modified polymer in the molten state, in order to facilitate a substantially homogeneous dispersion. When maleated polypropylene is the polar-modified hydrophobic polymer, the reaction may occur at about 160-225°C, preferably about 175-200°C.

The hydrophilic polymer reaction product is a polymer having increased hydrophilicity compared to both the hydrophobic polymer and the polar-modified hydrophobic polymer (which have advancing water contact angles greater than about 80 and sometimes about 90 or greater). Generally, the hydrophilic polymer reaction product has an advancing water contact angle less than about 80, preferably less than about 70, more preferably less than about 60, and in some instances less than about 50. Lower water contact angles for a material indicate greater hydrophilicity, and a greater tendency for water to wet the material.

It has also been discovered that, when polyglycols are used as the reactive hydrophilic material, higher molecular weight polyglycols cause a greater enhancement of hydrophilicity. When a polyglycol is used, its weight average molecular weight should be at least about 500, preferably at least about 1000, more preferably at least about 1500, and most preferably at least about 2000. Suitable JEFFAMINE® polyglycols, available from Huntsman Chemical Co., include those sold under the names M-600, M-1000, M-2005, ED-900 and ED-2003. These polyglycols differ in molecular weight and the number of amine groups per molecule. The JEFFAMINE® "M" series polyglycols are monoamines, while the "ED" series polyglycols are diamines. The numbers following the "M" and "ED" notations indicate weight average molecular weight.

Once formed, the hydrophilic polymer reaction product can be converted into a fibrous nonwoven web using a conventional melt spinning process. The resulting nonwoven web is of a durable hydrophilic character, meaning that the hydrophilicity cannot be washed away or otherwise easily removed. It is presently preferred to form the hydrophilic polymer reaction product, using the techniques described above, before forming the polymer into a nonwoven web. However, it is also contemplated that a nonwoven web may be formed before carrying out one or both reaction steps.

For instance, a nonwoven web may be formed from a polar-modified hydrophobic polymer, such as maleated polypropylene, after which the web can be surface-reacted with a hydrophilic material using a peroxide catalyst and a solution application. Similarly, a nonwoven web may initially be formed of a hydrophobic polymer, after which the web is surface-grafted with a polar monomer using catalyst and a solution application,

and then further reacted with a hydrophilic material. Other techniques for forming a hydrophilic nonwoven web by reacting a hydrophobic polymer, a polar material, and a hydrophilic material are also considered to be within the scope of the invention. For instance, a polar-modified polymer such as maleated polypropylene or polyethylene can be blended with a hydrophilic material such as polyethylene glycol or amine-terminated polyethylene oxide. The blend can then be spun into a nonwoven web, with the spinning conditions being controlled to assure a sufficient level of reaction between the hydrophilic material and the polar modified hydrophobic polymer, especially at the fiber surfaces.

In another embodiment, the hydrophilic polymer reaction product can be blended with a quantity of unmodified hydrophobic polymer (for example, an unmodified polyolefin such as polypropylene or polyethylene) to produce a blend having improved (blended) hydrophilic properties. The blend can then be spun into a nonwoven web. The blend composition may contain anywhere from about 2-100% of the hydrophilic polymer reaction product, depending on the level of hydrophilicity needed.

EXAMPLES 1-74

In the following examples, several maleated polyolefins were chemically reacted with polyolefin glycol materials using a Haake Rheocord 9000 batch mixer as the reaction chamber. The mixer was outfitted with twin blades and electric heating. For each example, a mixture of the maleated polyolefin and polyolefin glycol totaling 50 grams was placed in the batch mixer.

The maleated polyolefin was added to the mixer in the form of pellets. If the polyglycols were in liquid form, a syringe was used to add them. If the polyglycols were solid, they were added along with the maleated polyolefin. The batch mixer was set at 190°C, and the reaction was allowed to proceed for 10 minutes to form a hydrophilic polymer reaction product. After 10 minutes, samples of reaction product were collected from the batch mixer for analysis.

From the samples, films were pressed. Two separate films were pressed from each sample. Mylar sheets were used to prevent the resin blend from sticking to the film press. The film press was set at 190°C and 10000 psi for 1 minute. Then, water contact

angle measurements of those films were performed with a NRL Contact Angle Goniometer, Model 100-00, available from Rame-Hart, Inc.

The NRL Contact Angle Goniometer is a small, optical-bench type device incorporating an internal protractor-readout calibrated in 1-degree increments. Its low-power microscope produces a sharply-defined image of the water drop specimen, which is observed as a silhouette. A specimen supporting stage permits the specimen to be easily aligned with the two independently-rotatable crosshairs within the microscope and is calibrated on both horizontal and vertical axes in 0.02mm divisions. The variable intensity illuminator can be adjusted to allow for optimal illumination to be achieved. For these examples, a video camera was used to capture the image for display on a 14-inch monitor, allowing easy reading.

Three drops of water were placed onto each film sample, and the contact angles on both sides of the water droplets were recorded. These values were then averaged to give an average unwashed film contact angle. After measuring the contact angles, the films were washed off thoroughly with distilled water, and contact angles were measured again by the same method. This presumably washes away any excess residues or unreacted polyglycols on the surface of the resin that might affect the contact angle measurement.

Following is a list of the maleated polyolefins and polyglycols used for these experiments, as well as other ingredients.

Maleated Polyolefins

1. EXXELOR® 1015, described previously, polypropylene with 0.4% by weight maleic anhydride.
2. POLYBOND® 3150, described previously, polypropylene with 0.7% by weight maleic anhydride.
3. POLYBOND® 3200, described previously, polypropylene with 1.0% by weight maleic anhydride.
4. POLYBOND® 3009, a maleated polyethylene available from Uniroyal, having a melt flow rate (190°C) of 5 grams/10 min. and containing 1.0% by weight grafted maleic anhydride.
5. POLYBOND® 3002, a maleated polypropylene available from Uniroyal, having a melt flow rate (230°C) of 7 grams/10 min. and containing 0.2% by weight grafted maleic anhydride.
6. DOW S-1775, a maleated polyethylene available from Dow Chemical Co. containing 1.2% by weight maleic anhydride.

7. MP 660, a maleated polypropylene available from Aristech Chemical Co. containing 0.4% by weight maleic anhydride.

Polyglycols

1. JEFFAMINE® M-600, described previously, a monoamine polyglycol having a molecular weight of 600.
2. JEFFAMINE® M-1000, described previously, a monoamine polyglycol having a molecular weight of 1000.
3. JEFFAMINE® M-2005, described previously, a monoamine polyglycol having a molecular weight of 2005.
4. JEFFAMINE® ED-900, described previously, a diamine polyglycol having a molecular weight of 900.
5. JEFFAMINE® ED-2003, described previously, a diamine polyglycol having a molecular weight of 2003.
6. Polyethylene glycol, having a molecular weight of 2000, available from Aldrich Chemical Co.
7. Polyethylene glycol, having a molecular weight of 900, available from Aldrich Chemical Co.

Other Ingredients

1. EXXON 3445, a polypropylene homopolymer (not maleated or otherwise modified), used in some of the control Examples.
2. Masil SF-19, an ethoxylated ditriloxyane internal surfactant available from PPG Industries, used in some of the control Examples.
3. PEG 400 MO, a distearate internal surfactant available from PPG Industries, used in some of the control Examples.
4. Titanium propoxide, an esterification catalyst available from Aldrich Chemical Co. which can be used to aid the reaction between a polar functional polyolefin and a hydrophilic modifier.

The maleated polyolefins were reacted with varying amounts of the different polyglycols. The following Table 1 summarizes the water contact angles obtained for each Example.

Table 1: Contact Angle Measurements (Degrees)

Example		Sample 1	Sample 2	Average
1 EXXELOR 1015 4% JEFFAMINE M-1000	UNWASHED	60 60 59 59 65 67	62 60 57 58 65 67	61.6
	WASHED	79 81 78 75 74 72	73 77 72 75 73 75	75.3
2 EXXELOR 1015 8% JEFFAMINE M-1000	UNWASHED	65 66 75 75 72 75	70 67 63 57 72 72	69.1
	WASHED	83 83 77 78 75 75	74 72 81 78 72 71	76.6

Example		Sample 1	Sample 2	Average
3 EXXELOR 1015 16% JEFFAMINE M-1000	UNWASHED WASHED	63 63 57 54 55 54 73 67 73 70 70 63	48 49 42 43 44 46 67 65 60 65 67 63	51.5 66.9
4 EXXELOR 1015 4% JEFFAMINE M-2005	UNWASHED WASHED	64 63 62 60 61 60 80 82 75 76 78 78	71 74 64 64 63 65 68 64 70 72 74 75	64.3 74.3
5 EXXELOR 1015 8% JEFFAMINE M-2005	UNWASHED WASHED	56 55 52 53 53 53 68 66 65 64 67 67	42 40 40 41 37 41 73 71 70 66 68 68	46.9 67.8
6 EXXELOR 1015 16% JEFFAMINE M-2005	UNWASHED WASHED	42 44 42 44 44 45 57 54 57 56 53 51	45 45 42 43 41 41 65 59 54 53 59 57	43.2 56.3
7 EXXELOR 1015 4% JEFFAMINE M-600	UNWASHED WASHED	40 40 41 42 40 39 75 73 76 75 73 68	53 57 62 65 54 58 78 81 78 78 77 78	49.3 75.8
8 EXXELOR 1015 8% JEFFAMINE M-600	UNWASHED WASHED	46 45 50 54 47 54 76 75 74 77 76 75	57 54 54 54 55 58 75 78 80 80 75 74	52.3 76.3
9 EXXELOR 1015 16% JEFFAMINE M-600	UNWASHED WASHED	57 58 57 58 62 60 75 78 74 75 75 71	45 40 43 42 41 40 77 74 75 77 78 79	50.3 75.7
10 POLYBOND 3009 5% JEFFAMINE M-2005	UNWASHED WASHED	67 68 74 72 68 73 68 62 64 62 63 59	75 74 67 72 64 67 63 68 64 64 65 62	70.1 63.7
11 POLYBOND 3009 10% JEFFAMINE M-2005	UNWASHED WASHED	75 74 65 64 65 66 67 68 67 68 67 68	53 55 54 53 52 53 75 73 72 71 69 69	60.8 69.5
12 POLYBOND 3200 4% JEFFAMINE M-1000	UNWASHED WASHED	66 62 62 62 62 64 66 66 65 66 66 66	60 62 62 65 60 65 63 63 66 66 62 63	62.7 64.8
13 POLYBOND 3200 8% JEFFAMINE M-1000	UNWASHED WASHED	52 54 56 53 55 53 67 66 65 64 64 62	52 53 54 54 56 54 62 64 66 68 67 71	53.8 65.5
14 POLYBOND 3200 16% JEFFAMINE M-1000	UNWASHED WASHED	45 44 47 43 44 43 63 64 63 63 63 62	45 42 41 40 42 42 65 62 61 59 62 60	43.2 62.3
15 POLYBOND 3200 4% JEFFAMINE M-2005	UNWASHED WASHED	60 60 60 64 60 58 69 71 74 74 69 72	55 55 55 53 56 55 69 71 66 70 67 68	57.6 70.0
16 POLYBOND 3200 8% JEFFAMINE M-2005	UNWASHED WASHED	58 57 60 56 61 61 67 64 61 64 63 61	60 60 59 60 59 61 60 59 61 59 65 65	59.3 62.4
17 POLYBOND 3200 16% JEFFAMINE M-2005	UNWASHED WASHED	62 62 60 59 59 60 74 71 72 72 71 69	60 56 55 54 53 52 70 72 67 66 63 62	57.7 69.1
18 POLYBOND 3200 4% JEFFAMINE M-600	UNWASHED WASHED	63 62 60 58 57 60 72 71 71 70 71 70	65 69 68 65 64 66 77 74 72 70 70 68	63.1 71.3
19 POLYBOND 3200 8% JEFFAMINE M-600	UNWASHED WASHED	56 58 54 53 58 59 73 73 72 71 71 70	66 67 62 62 61 61 72 71 75 76 72 74	59.8 72.5
20 POLYBOND 3200 16% JEFFAMINE M-600	UNWASHED WASHED	60 60 57 59 60 61 70 71 71 74 69 71	67 65 65 60 60 62 70 75 71 71 75 75	61.3 71.9
21 POLYBOND 3200 4% JEFFAMINE ED-2003	UNWASHED WASHED	55 53 65 67 65 67 72 69 71 69 72 70	57 59 64 67 64 65 65 67 65 66 64 67	62.3 68.1

Example		Sample 1	Sample 2	Average
22 POLYBOND 3200 8% JEFFAMINE ED-2003	UNWASHED WASHED	67 65 65 67 67 65 70 71 71 73 72 70	68 68 67 66 66 68 70 69 71 69 72 74	66.6 71.0
23 (NO RESULTS)				
24 POLYBOND 3200 4% JEFFAMINE ED-900	UNWASHED WASHED	65 66 68 69 68 69 70 71 75 74 70 74	66 64 65 61 66 64 75 77 75 73 70 70	65.9 72.8
25 POLYBOND 3200 8% JEFFAMINE ED-900	UNWASHED WASHED	53 50 52 49 44 47 64 62 62 61 62 60	60 61 54 57 58 54 63 67 64 64 63 65	53.3 63.1
26 POLYBOND 3200 16% JEFFAMINE ED-900	UNWASHED WASHED	52 50 48 50 47 49 63 60 59 62 63 65	45 49 41 39 42 40 52 50 53 51 55 51	46.0 57.0
27 EXXELOR 1015 4% JEFFAMINE ED-2003	UNWASHED WASHED	62 64 62 63 64 65 72 69 71 69 72 70	46 48 49 53 51 52 60 63 63 64 63 63	56.6 66.6
28 EXXELOR 1015 8% JEFFAMINE ED-2003	UNWASHED WASHED	63 62 62 61 59 61 68 65 64 65 67 66	47 50 50 52 48 53 63 64 62 63 63 62	55.7 64.3
29 EXXELOR 1015 16% JEFFAMINE ED-2003	UNWASHED WASHED	44 40 44 41 42 41 60 57 61 56 50 52	44 41 40 42 42 41 53 55 54 58 58 58	41.8 56.0
30 EXXELOR 1015 4% JEFFAMINE ED-900	UNWASHED WASHED	64 65 66 65 65 67 72 72 73 71 72 68	62 63 62 59 62 60 69 70 68 68 69 68	63.3 70.0
31 EXXELOR 1015 8% JEFFAMINE ED-900	UNWASHED WASHED	47 46 50 48 46 49 65 62 62 60 61 60	45 44 43 47 46 41 63 61 61 59 61 59	46.0 61.2
32 EXXELOR 1015 16% JEFFAMINE ED-900	UNWASHED WASHED	63 64 64 68 62 66 72 73 75 75 73 72	43 45 42 41 39 42 63 64 64 65 64 64	53.3 68.7
33 POLYBOND 3009 5% JEFFAMINE M-600	UNWASHED WASHED	55 58 52 56 60 55 63 63 63 64 64 63	62 65 64 64 64 62 71 72 68 64 66 66	59.8 65.6
34 POLYBOND 3009 10% JEFFAMINE M-600	UNWASHED WASHED	62 65 63 63 61 66 65 67 69 70 64 65	62 63 63 66 65 62 67 65 63 62 65 61	63.4 65.3
35 POLYBOND 3009 5% JEFFAMINE M-1000	UNWASHED WASHED	52 53 58 56 54 56 60 65 58 56 62 59	59 57 59 59 59 61 62 59 65 67 65 61	56.9 61.6
36 POLYBOND 3009 10% JEFFAMINE M-1000	UNWASHED WASHED	57 57 57 55 57 58 65 63 60 63 65 62	56 54 54 52 54 49 63 60 60 57 62 59	55.0 61.6
37 POLYBOND 3150 4% JEFFAMINE M-1000	UNWASHED WASHED	42 44 43 41 44 43 60 60 64 65 60 62	44 44 40 42 47 48 62 60 63 61 61 61	43.5 61.6
38 POLYBOND 3150 8% JEFFAMINE M-1000	UNWASHED WASHED	63 60 56 54 54 54 63 64 60 59 62 62	50 49 53 51 50 48 62 63 58 57 62 59	53.5 60.9
39 POLYBOND 3150 16% JEFFAMINE M-1000	UNWASHED WASHED	51 54 56 57 56 54 71 65 70 66 67 66	54 51 55 54 53 51 74 70 73 71 66 70	53.8 69.1
40 POLYBOND 3150 4% JEFFAMINE M-2005	UNWASHED WASHED	49 51 48 46 52 48 65 63 62 60 57 60	58 55 50 47 49 46 61 62 61 60 60 60	49.9 60.9

Example		Sample 1	Sample 2	Average
41 POLYBOND 3150 8% JEFFAMINE M-2005	UNWASHED WASHED	49 50 53 51 52 51 57 56 56 58 55 56	42 45 43 41 48 44 54 52 58 53 55 52	47.4 55.2
42 POLYBOND 3150 16% JEFFAMINE M-2005	UNWASHED WASHED	42 43 42 38 46 43 58 56 55 54 51 48	43 40 46 44 49 48 63 61 61 60 59 62	43.7 57.3
43 POLYBOND 3150 4% JEFFAMINE M-600	UNWASHED WASHED	65 65 66 64 65 68 70 69 76 75 76 78	73 71 66 65 64 66 75 75 78 76 77 76	66.5 75.1
44 POLYBOND 3150 8% JEFFAMINE M-600	UNWASHED WASHED	65 62 64 63 63 61 67 65 69 68 73 71	60 63 63 60 64 62 72 73 66 64 69 69	62.5 68.8
45 POLYBOND 3150 16% JEFFAMINE M-600	UNWASHED WASHED	54 53 57 59 60 59 75 73 73 74 72 74	48 46 50 46 51 47 72 72 69 73 75 73	52.5 72.9
46 POLYBOND 3150 4% JEFFAMINE ED-2003	UNWASHED WASHED	66 65 67 64 62 63 68 64 64 63 65 63	67 66 66 69 67 68 66 66 67 63 66 64	65.8 64.9
47 POLYBOND 3150 8% JEFFAMINE ED-2003	UNWASHED WASHED	58 57 60 58 57 58 64 65 62 63 63 64	59 58 60 58 57 56 64 62 63 63 64 64	58.0 63.4
48 POLYBOND 3150 16% JEFFAMINE ED-2003	UNWASHED WASHED	--- --- --- --- --- --- 42 40 45 42 42 42	--- --- --- --- --- --- 47 45 46 46 43 43	----- 43.6
49 POLYBOND 3150 4% JEFFAMINE ED-900	UNWASHED WASHED	54 52 52 50 49 51 59 58 57 59 59 58	49 48 52 50 48 50 59 58 60 60 59 57	50.4 58.6
50 POLYBOND 3150 8% JEFFAMINE ED-900	UNWASHED WASHED	48 49 46 48 47 47 56 54 55 55 54 54	50 49 48 45 45 46 56 58 59 59 55 56	47.3 55.9
51 POLYBOND 3150 16% JEFFAMINE ED-900	UNWASHED WASHED	26 26 29 28 27 27 55 55 56 54 54 54	60 63 63 60 61 62 67 67 64 65 64 66	44.3 60.1
52 POLYBOND 3150 12% JEFFAMINE M-2005	UNWASHED WASHED	37 37 36 38 35 39 50 52 50 48 49 50	39 40 38 39 38 38 51 49 52 50 52 50	37.8 50.3
53 POLYBOND 3150 20% JEFFAMINE M-2005	UNWASHED WASHED	--- --- --- --- --- --- 45 47 48 49 45 45	--- --- --- --- --- --- 44 43 40 41 42 40	----- 44.1
54 POLYBOND 3150 24% JEFFAMINE M-2005	UNWASHED WASHED	--- --- --- --- --- --- 42 43 43 42 41 43	--- --- --- --- --- --- 40 42 43 42 42 43	----- 42.2
55 PP 3445 2% SF-19	UNWASHED WASHED	--- --- --- --- --- --- 87 90 88 88 85 87	--- --- --- --- --- --- 85 88 88 88 86 89	----- 87.4
56 PP 3445 2% SF-19 2% EXXELOR 1015	UNWASHED WASHED	--- --- --- --- --- --- 90 89 92 89 90 88	--- --- --- --- --- --- 87 88 87 87 88 89	----- 88.7
57 PP 3445 2% SF-19 2% POLYBOND 3150	UNWASHED WASHED	--- --- --- --- --- --- 90 90 88 89 89 92	--- --- --- --- --- --- 92 91 91 89 91 90	----- 90.2
58 PP 3445 2% PEG400 MO	UNWASHED WASHED	64 66 67 65 62 60 94 91 92 92 93 92	60 58 62 60 58 60 89 91 93 91 92 90	61.8 91.7

Example		Sample 1	Sample 2	Average
59 PP 3445 2% PEG400 MO 2% EXXELOR 1015	UNWASHED WASHED	54 53 55 52 52 55 92 92 91 90 92 90	48 50 49 51 48 50 90 90 90 92 92 91	51.4 91.0
60 PP 3445 2% PEG400 MO 2% POLYBOND 3150	UNWASHED WASHED	52 50 52 51 48 49 90 88 90 89 90 88	57 55 54 53 55 53 90 90 94 92 91 91	52.4 90.3
61 EXXELOR 1015 20 % JEFFAMINE M-2005	UNWASHED WASHED	--- --- --- --- --- 54 54 53 55 51 55	--- --- --- --- --- 52 52 54 54 53 53	--- 53.3
62 EXXELOR 1015 24% JEFFAMINE M-2005	UNWASHED WASHED	--- --- --- --- --- 50 51 50 51 49 51	--- --- --- --- --- 49 50 50 48 51 49	--- 49.9
63 EXXELOR 1015 20% JEFFAMINE ED-2003	UNWASHED WASHED	--- --- --- --- --- 52 52 50 50 51 50	--- --- --- --- --- 47 49 48 48 47 48	--- 49.3
64 EXXELOR 1015 24% JEFFAMINE ED-2003	UNWASHED WASHED	--- --- --- --- --- 50 50 50 50 50 49	--- --- --- --- --- 47 47 48 47 47 47	--- 48.5
65 POLYBOND 3200 20% JEFFAMINE ED-900	UNWASHED WASHED	42 40 40 42 43 44 57 56 55 55 58 55	42 41 43 43 42 41 62 63 62 62 62 63	41.9 59.2
66 POLYBOND 3200 24% JEFFAMINE ED-900	UNWASHED WASHED	40 40 42 43 43 42 58 57 58 60 58 59	42 43 44 43 43 42 63 63 62 61 62 63	42.3 60.3
67 POLYBOND 3150 4% JEFFAMINE M-2005	UNWASHED WASHED	43 43 48 47 45 45 60 55 54 55 52 53	47 52 48 52 48 52 60 58 60 57 58 58	47.5 56.7
68 POLYBOND 3150 8% JEFFAMINE M-2005	UNWASHED WASHED	40 44 42 44 42 43 54 50 55 55 53 50	40 44 44 45 44 40 54 52 55 52 54 50	42.7 52.8
69 POLYBOND 3150 16% JEFFAMINE M-2005	UNWASHED WASHED	30 35 33 35 33 31 53 53 53 53 55 51	40 40 40 41 40 41 55 55 58 54 54 56	36.6 54.2
70 POLYBOND 3150 12% JEFFAMINE M-2005	UNWASHED WASHED	43 43 42 45 43 44 55 51 56 50 53 53	41 41 45 44 43 43 53 53 55 55 54 53	43.1 53.4
71 POLYBOND 3150 4% JEFFAMINE ED-900	UNWASHED WASHED	65 63 66 62 63 64 67 63 65 65 68 67	58 56 58 56 62 62 65 60 64 60 62 63	61.3 64.1
72 POLYBOND 3150 8% JEFFAMINE ED-900	UNWASHED WASHED	52 50 51 51 51 50 62 57 62 57 61 56	56 54 54 53 56 52 62 58 62 59 62 61	52.5 59.9
73 POLYBOND 3002 4% JEFFAMINE M-2000	UNWASHED WASHED	47 48 45 50 47 46 57 56 54 54 57 56	47 50 48 48 49 49 56 58 55 55 54 55	47.8 55.6
74 POLYBOND 3002 8% JEFFAMINE M-2000	UNWASHED WASHED	47 45 43 41 46 43 52 51 55 53 53 52	42 40 46 48 48 44 53 52 54 53 54 54	44.4 53.0

The results of Examples 1-74 are compared in various ways by graphing (Figs. 1-8). Figs. 1 and 2 illustrate the effects of different maleic anhydride levels in polypropylene and different polyglycol levels for a JEFFAMINE® polyglycol, M-2005, after

washing. For all three levels of maleic anhydride (0.4%, 0.7% and 1.0%), higher levels of polyglycol (20% and 24% by weight) resulted in lower contact angles. In general, the polypropylene with 0.7% by weight maleic anhydride resulted in better hydrophilicity (lower contact angles) than the polypropylenes with 0.4% and 1.0% by weight maleic anhydride.

Fig. 3 illustrates the effects of using polyglycols of different molecular weight and different percentage levels, for maleated polypropylene containing 0.4% by weight maleic anhydride. The contact angles were lowered (indicating better hydrophilicity) as 1) the molecular weight of polyglycol was raised, and 2) the amount of polyglycol was increased.

Fig. 4 illustrates the effects of using the lowest molecular weight polyglycol (M-600) in different amounts, with two levels of anhydride-grafted polypropylene (0.4% and 1.0% by weight). Better contact angles were achieved with the higher level of anhydride modification. Yet there was little change in contact angles as the polyglycol levels were varied between 4% and 16% by weight.

Fig. 5 illustrates the effect of washing on samples made using all three of the JEFFAMINE "M"-series polyglycols, at three levels of polyglycol, and polypropylene grafted with 0.7% by weight maleic anhydride. The washing caused the contact angles to increase, but not enough to render the samples hydrophobic. The washing may have removed unreacted monomer and impurities, but did not remove the chemically imposed hydrophilicity resulting from the chemical reaction between the maleated polypropylene and the hydrophilic materials.

Fig. 6 illustrates that contact angles on reaction products from different trials are quite reproducible for different samples prepared the same way, using the same ingredients.

Fig. 7 illustrates the effect of reacting different levels of monoamine polyglycol, with molecular weight of 2005, with maleated polypropylene containing 0.7% by weight maleic anhydride. In general, the contact angles decreased as the polyglycol level was raised. However, Fig. 8 illustrates that the contact angle is roughly independent of polyglycol level when a diamine polyglycol having a molecular weight of 900 is used.

EXAMPLE 75

A maleated polyethylene, manufactured by Dow Chemical Co. under the name S-1775, purportedly having a 1.2 wt% maleic anhydride content was mixed with 5 wt% of poly(ethylene glycol), molecular weight 2,000, in the above mentioned mixer at 190 degrees C for 10 min. The contact angle of the pressed films from this compound was measured as 48 degrees before washing, and 59 degrees after washing the film with ample water and drying. The original S-1775 resin had a contact angle of 84 degrees.

EXAMPLE 76

A maleated polypropylene, manufactured by Exxon Chemical Co. under the name of Exxelor 1015, having a claimed 0.4 wt% of maleic anhydride content was mixed with 4 wt% of poly(ethylene glycol), molecular weight 2,000, in the above mentioned mixer at 200 degrees C for 10 min. The contact angle of the pressed films from this compound was measured as 51 degrees before washing, and 69 degrees after washing the film with ample water and drying.

EXAMPLE 77

A maleated polypropylene, manufactured by Aristech Chemical Co. under the name of MP660, having a claimed 0.4 wt% of maleic anhydride content was mixed with 4 wt% of poly(ethylene glycol), molecular weight 900, together with 0.2 wt.% of esterification catalyst titanium propoxide obtained from Aldrich Chemical Co., in the above mentioned mixer at 200 degrees C for 10 min. The contact angle of the pressed films from this compound was measured as 38 degrees before washing, and 56 degrees after washing the film with ample water and drying.

EXAMPLES 78-91

Using the techniques of Examples 75-77, fourteen additional compositions were prepared and tested. The results are summarized in Table 2 below.

Table 2: Contact Angles (Degrees)

Example	Maleated Polymer	Maleated Polymer Brand	Hydrophilic Material	Molecular Weight	Weight Percent	Other Additives	Contact Angle	
							Before Wash	After Wash
78	Polyethylene	Dow S-1775	-----	----	----	----	84	N.A.
79	Polyethylene	Dow S-1775	Polyethylene glycol	2000	5	----	48	59
80	Polyethylene	Dow S-1775	Polyethylene glycol	2000	10	----	53	N.A.
81	Polypropylene	Aristech MP660	-----	----	----	----	79	N.A.
82	Polypropylene	Aristech MP660	Polyethylene glycol	900	2	----	43	60
83	Polypropylene	Aristech MP660	Polyethylene glycol	900	4	----	49	63
84	Polypropylene	Aristech MP660	Polyethylene glycol	900	6	----	51	70
85	Polypropylene	Aristech MP660	Polyethylene glycol	900	4	Ti Catalyst	38	56
86	Polypropylene	Aristech MP660	Polyethylene glycol	300	4	Ti Catalyst	64	68
87	Polypropylene	Aristech MP660	Polyethylene glycol	8000	10	----	48	60
88	Polypropylene	EXXELOR * 1015	Polyethylene glycol	2000	4	----	51	69
89	Polypropylene	EXXELOR * 1015	Polyethylene glycol	2000	8	----	59	71
90	Polypropylene	EXXELOR * 1015	JEFFAMINE *M-2000	2000	4	----	51	62
91	Polypropylene	EXXELOR * 1015	JEFFAMINE *M-2000	2000	8	----	46	56

While the embodiments of the invention described herein are presently considered preferred, various modifications and improvements can be made without departing from the spirit and scope of the invention. The scope of the invention is indicated by the appended claims, and all changes that fall within the meaning and range of equivalents are intended to be embraced therein.

WE CLAIM:

- I. A nonwoven web of fibers, comprising the reaction product of:
 - a hydrophobic polymer material having a water contact angle of at least 80 degrees, measured using ASTM D5946-96;
 - a polar material chemically reacted with the hydrophobic polymer to form a modified polymer containing at least about 0.1% by weight of the polar material; and
 - a hydrophilic material chemically reacted with the modified polymer to form a hydrophilic polymer reaction product having a lower water contact angle than the hydrophobic polymer.
2. The nonwoven web of Claim 1, wherein the hydrophobic polymer comprises a material selected from the group consisting of polypropylene, polyethylene, ethylene copolymers with C₃-C₂₀ α -olefins, propylene copolymers with ethylene or C₄-C₂₀ α -olefins, butene copolymers with ethylene, propylene, or C₃-C₂₀ α -olefins, polyvinyl chloride, polyesters, polyfluorocarbons, hydrophobic polyurethane, polystyrene, acrylic resins, and combinations thereof.
3. The nonwoven web of Claim 1, wherein the hydrophobic polymer comprises a polyolefin.
4. The nonwoven web of Claim 3, wherein the polyolefin comprises a material selected from the group consisting of polyethylenes, polypropylenes, copolymers thereof, and blends thereof.
5. The nonwoven web of Claim 4, wherein the polyolefin comprises a polypropylene.
6. The nonwoven web of Claim 4, wherein the polyolefin comprises a polyethylene.

7. The nonwoven web of Claim 1, wherein the polar material comprises a material selected from the group consisting of anhydrides, carboxylic acid derivatives thereof, and combinations of the foregoing.

8. The nonwoven web of Claim 1, wherein the polar material comprises a material selected from the group consisting of maleic anhydride, carboxylic acid derivatives thereof, and combinations of the foregoing.

9. The nonwoven web of Claim 1, wherein the hydrophilic material comprises a material selected from the group consisting of polyglycols, polyoxides, and combinations thereof.

10. The nonwoven web of Claim 9, wherein the hydrophilic material comprises a material selected from the group consisting of polyolefin glycols, polyolefin oxides, and combinations thereof.

11. The nonwoven web of Claim 10, wherein the hydrophilic material comprises a material selected from the group consisting of polyethylene glycol, polyethylene oxide, and combinations thereof.

12. The nonwoven web of Claim 10, wherein the hydrophilic material comprises a material selected from the group consisting of polypropylene glycol, polypropylene oxide, and combinations thereof.

13. The nonwoven web of Claim 10, wherein the hydrophilic material comprises a polyolefin glycol having an amine linkage.

14. The nonwoven web of Claim 13, wherein the amine linkage comprises a monoamine.

15. The nonwoven web of Claim 13, wherein the amine linkage comprises a diamine.

16. The nonwoven web of Claim 1, wherein the modified polymer comprises about 0.1-3.0% by weight of the polar material.

17. The nonwoven web of Claim 1, wherein the modified polymer comprises about 0.4-1.0% by weight of the polar material.

18. The nonwoven web of Claim 1, wherein the modified polymer comprises about 0.6-0.8% by weight of the polar material.

19. The nonwoven web of Claim 1, wherein the hydrophilic material comprises about 1-35% by weight of the hydrophilic polymer reaction product having increased hydrophilic properties.

20. The nonwoven web of Claim 19, wherein the hydrophilic material comprises about 4-25% by weight of the hydrophilic polymer reaction product.

21. The nonwoven web of Claim 19, wherein the hydrophilic material comprises about 8-20% by weight of the hydrophilic polymer reaction product.

22. A nonwoven web of fibers, comprising the reaction product of:
a hydrophobic polymer material having a water contact angle greater than about 80 degrees, measured using ASTM D5946-96;
a polar material chemically reacted with the hydrophobic polymer to form a modified polymer containing at least about 0.1% by weight of the polar material; and
a hydrophilic material chemically reacted with the modified polymer to form a hydrophilic polymer reaction product having a water contact angle less than about 80 degrees.

23. The nonwoven web of Claim 22, wherein the hydrophobic polymer has an initial water contact angle of at least about 90 degrees.
24. The nonwoven web of Claim 22, wherein the hydrophilic polymer reaction product has a water contact angle less than about 70 degrees.
25. The nonwoven web of Claim 22, wherein the hydrophilic polymer reaction product has a water contact angle less than about 60 degrees.
26. The nonwoven web of Claim 22, wherein the hydrophilic polymer reaction product has a water contact angle less than about 50 degrees.
27. The nonwoven web of Claim 22, comprising a spunbond web.
28. The nonwoven web of Claim 22, comprising a meltblown web.
29. The nonwoven web of Claim 22, comprising a bonded carded web.
30. A nonwoven web of fibers, comprising a maleated polyolefin material chemically reacted with a hydrophilic material to impart durable hydrophilic properties to the nonwoven web.
31. The nonwoven web of Claim 30, wherein the maleated polyolefin comprises a polyolefin reacted with maleic anhydride.
32. The nonwoven web of Claim 30, wherein the maleated polyolefin comprises an ethylene polymer.
33. The nonwoven web of Claim 30, wherein the maleated polyolefin comprises a propylene polymer.

34. The nonwoven web of Claim 30, wherein the hydrophilic polymer comprises a polyglycol or polyoxide.

35. The nonwoven web of Claim 30, wherein the hydrophilic polymer comprises a polyolefin glycol.

36. The nonwoven web of Claim 35, wherein the polyolefin glycol includes at least one amine group.

37. The nonwoven web of Claim 30, wherein the hydrophilic material has a weight average molecular weight of at least about 500.

38. The nonwoven web of Claim 30, wherein the hydrophilic material has a weight average molecular weight of at least about 1000.

39. The nonwoven web of Claim 30, wherein the hydrophilic material has a weight average molecular weight of at least about 1500.

40. The nonwoven web of Claim 30, wherein the hydrophilic material has a weight average molecular weight of at least about 2000.

41. An absorbent product comprising the nonwoven web of Claim 1.

42. Apparel comprising the nonwoven web of Claim 1.

43. A diaper comprising the nonwoven web of Claim 1.

44. A tampon comprising the nonwoven web of Claim 1.

- 45. A medical garment comprising the nonwoven web of Claim 1.
- 46. A surgical glove comprising the nonwoven web of Claim 1.
- 47. A cap comprising the nonwoven web of Claim 1.
- 48. An apron comprising the nonwoven web of Claim 1.
- 49. A sterilization wrap comprising the nonwoven web of Claim 1.

AMENDED CLAIMS

[received by the International Bureau on 28 January 2000 (28.01.00);
original claims 1, 22 and 30 amended; remaining claims unchanged (6 pages)]

1. A nonwoven web of fibers, the fibers formed from a polymer or polymer blend comprising a hydrophilic polymer reaction product which is the reaction product of:

a hydrophobic polymer material having a water contact angle of at least 80 degrees, measured using ASTM D5946-96;

a polar material chemically reacted with the hydrophobic polymer to form a modified polymer containing at least about 0.1% by weight of the polar material; and

a hydrophilic material blended and chemically reacted with the modified polymer to form the hydrophilic polymer reaction product having a lower water contact angle than the hydrophobic polymer both before and after the hydrophilic polymer reaction product is washed with distilled water.

2. The nonwoven web of Claim 1, wherein the hydrophobic polymer comprises a material selected from the group consisting of polypropylene, polyethylene, ethylene copolymers with C₃-C₂₀ α -olefins, propylene copolymers with ethylene or C₄-C₂₀ α -olefins, butene copolymers with ethylene, propylene, or C₅-C₂₀ α -olefins, polyvinyl chloride, polyesters, polyfluorocarbons, hydrophobic polyurethane, polystyrene, acrylic resins, and combinations thereof.

3. The nonwoven web of Claim 1, wherein the hydrophobic polymer comprises a polyolefin.

4. The nonwoven web of Claim 3, wherein the polyolefin comprises a material selected from the group consisting of polyethylenes, polypropylenes, copolymers thereof, and blends thereof.

5. The nonwoven web of Claim 4, wherein the polyolefin comprises a polypropylene.

6. The nonwoven web of Claim 4, wherein the polyolefin comprises a polyethylene.

7. The nonwoven web of Claim 1, wherein the polar material comprises a material selected from the group consisting of anhydrides, carboxylic acid derivatives thereof, and combinations of the foregoing.

8. The nonwoven web of Claim 1, wherein the polar material comprises a material selected from the group consisting of maleic anhydride, carboxylic acid derivatives thereof, and combinations of the foregoing.

9. The nonwoven web of Claim 1, wherein the hydrophilic material comprises a material selected from the group consisting of polyglycols, polyoxides, and combinations thereof.

10. The nonwoven web of Claim 9, wherein the hydrophilic material comprises a material selected from the group consisting of polyolefin glycols, polyolefin oxides, and combinations thereof.

11. The nonwoven web of Claim 10, wherein the hydrophilic material comprises a material selected from the group consisting of polyethylene glycol, polyethylene oxide, and combinations thereof.

12. The nonwoven web of Claim 10, wherein the hydrophilic material comprises a material selected from the group consisting of polypropylene glycol, polypropylene oxide, and combinations thereof.

13. The nonwoven web of Claim 10, wherein the hydrophilic material comprises a polyolefin glycol having an amine linkage.

14. The nonwoven web of Claim 13, wherein the amine linkage comprises a monoamine.

15. The nonwoven web of Claim 13, wherein the amine linkage comprises a diamine.

16. The nonwoven web of Claim 1, wherein the modified polymer comprises about 0.1-3.0% by weight of the polar material.

17. The nonwoven web of Claim 1, wherein the modified polymer comprises about 0.4-1.0% by weight of the polar material.

18. The nonwoven web of Claim 1, wherein the modified polymer comprises about 0.6-0.8% by weight of the polar material.

19. The nonwoven web of Claim 1, wherein the hydrophilic material comprises about 1-35% by weight of the hydrophilic polymer reaction product having increased hydrophilic properties.

20. The nonwoven web of Claim 19, wherein the hydrophilic material comprises about 4-25% by weight of the hydrophilic polymer reaction product.

21. The nonwoven web of Claim 19, wherein the hydrophilic material comprises about 8-20% by weight of the hydrophilic polymer reaction product.

22. A nonwoven web of fibers, the fibers formed from a polymer or polymer blend comprising a hydrophilic polymer reaction product which is the reaction product of:

a hydrophobic polymer material having a water contact angle greater than about 80 degrees, measured using ASTM D5946-96;

a polar material chemically reacted with the hydrophobic polymer to form a modified polymer containing at least about 0.1% by weight of the polar material; and

a hydrophilic material blended and chemically reacted with the modified polymer to form the hydrophilic polymer reaction product having a water contact angle less than about 80 degrees both before and after the hydrophilic polymer reaction product is washed with distilled water.

23. The nonwoven web of Claim 22, wherein the hydrophobic polymer has an initial water contact angle of at least about 90 degrees.

24. The nonwoven web of Claim 22, wherein the hydrophilic polymer reaction product has a water contact angle less than about 70 degrees.

25. The nonwoven web of Claim 22, wherein the hydrophilic polymer reaction product has a water contact angle less than about 60 degrees.

26. The nonwoven web of Claim 22, wherein the hydrophilic polymer reaction product has a water contact angle less than about 50 degrees.

27. The nonwoven web of Claim 22, comprising a spunbond web.

28. The nonwoven web of Claim 22, comprising a meltblown web.

29. The nonwoven web of Claim 22, comprising a bonded carded web.

30. A nonwoven web of fibers, the fibers formed from a polymer or polymer blend comprising a maleated polyolefin material blended and chemically reacted with a hydrophilic material to impart durable hydrophilic properties to the nonwoven web that withstand washing with distilled water.

31. The nonwoven web of Claim 30, wherein the maleated polyolefin comprises a polyolefin reacted with maleic anhydride.

32. The nonwoven web of Claim 30, wherein the maleated polyolefin comprises an ethylene polymer.

33. The nonwoven web of Claim 30, wherein the maleated polyolefin comprises a propylene polymer.

34. The nonwoven web of Claim 30, wherein the hydrophilic polymer comprises a polyglycol or polyoxide.

35. The nonwoven web of Claim 30, wherein the hydrophilic polymer comprises a polyolefin glycol.

36. The nonwoven web of Claim 35, wherein the polyolefin glycol includes at least one amine group.

37. The nonwoven web of Claim 30, wherein the hydrophilic material has a weight average molecular weight of at least about 500.

38. The nonwoven web of Claim 30, wherein the hydrophilic material has a weight average molecular weight of at least about 1000.

39. The nonwoven web of Claim 30, wherein the hydrophilic material has a weight average molecular weight of at least about 1500.

40. The nonwoven web of Claim 30, wherein the hydrophilic material has a weight average molecular weight of at least about 2000.

41. An absorbent product comprising the nonwoven web of Claim 1.

42. Apparel comprising the nonwoven web of Claim 1.

43. A diaper comprising the nonwoven web of Claim 1.

44. A tampon comprising the nonwoven web of Claim 1.

45. A medical garment comprising the nonwoven web of Claim 1.

46. A surgical glove comprising the nonwoven web of Claim 1.

- 47. A cap comprising the nonwoven web of Claim 1.
- 48. An apron comprising the nonwoven web of Claim 1.
- 49. A sterilization wrap comprising the nonwoven web of Claim 1.

STATEMENT UNDER ARTICLE 19

Claims 1, 22 and 30 have been replaced with amended Claims 1, 22 and 30. Claims 2-21, 23-29 and 31-49 are unchanged in text, but changed in scope due to their dependence on amended Claims 1, 22 and 30.

The amended Claims 1, 22 and 30 differ from the original claims by reciting that the fibers are formed from a polymer or polymer blend comprising the hydrophilic polymer reaction products. The amended claims further recite that the hydrophilic material is blended and chemically reacted with the modified polymer to form the hydrophilic polymer reaction product. The effect of these amendments is to clearly indicate that the fibers are formed after the chemical reaction has been completed. Finally, the amended claims require that the reaction product has hydrophilic properties that cannot be washed away with distilled water.

Put another way, the Applicants' claims do not relate to prior art nonwoven webs which are formed using hydrophobic polymers, and then merely surface coated with a hydrophilic material. Instead, the fibers are formed after the hydrophobic polymer has been rendered hydrophilic. One advantage of Applicant's nonwoven webs is that the fibers contain the hydrophilic polymer reaction product all the way through. Because of this, the hydrophilic polymer reaction product cannot be washed away from the nonwoven web, and the hydrophilic characteristics are more durable. Prior art nonwoven webs which are merely surface treated with hydrophilic materials have less durability, because the coatings can be more easily washed away.

The amended Claims 1, 22 and 30 clearly distinguish the invention from the prior art. Neither European Publication 0,114,379, nor European Publication 0,634,424, discloses a nonwoven web of fibers having hydrophilic properties that cannot be washed away with distilled water. Neither reference suggests a hydrophilic polymer reaction product having a lower water contact angle than the hydrophobic polymer both before and after the reaction product is washed with distilled water, as recited in amended Claim 1. Similarly, neither reference suggests a water contact angle less than 80 degrees before and after the hydrophilic polymer reaction product is washed, as recited in amended Claim 22, or hydrophilic properties that withstand washing as recited in amended Claim 30. As explained on page 12 of Applicant's specification, the washing test requires sufficiently thorough washing to remove any excess residues or unreacted polyglycols that might affect the water contact angle measurement.

Applicant respectfully requests entry of a favorable International Preliminary Examination Report.

1/8

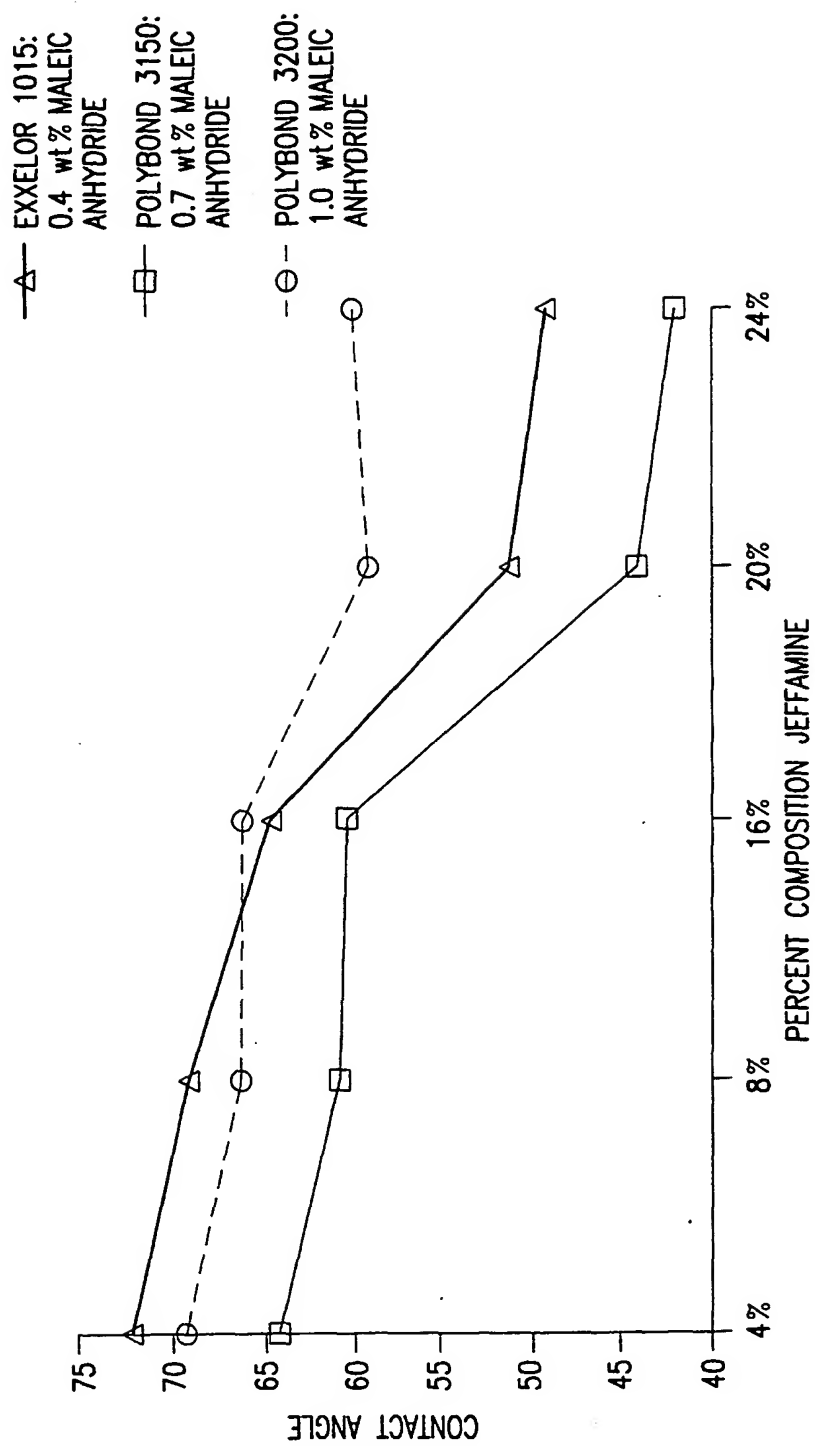


FIG.1

2/8

—△— 4% M-2005
—□— 8% M-2005
—○— 16% M-2005

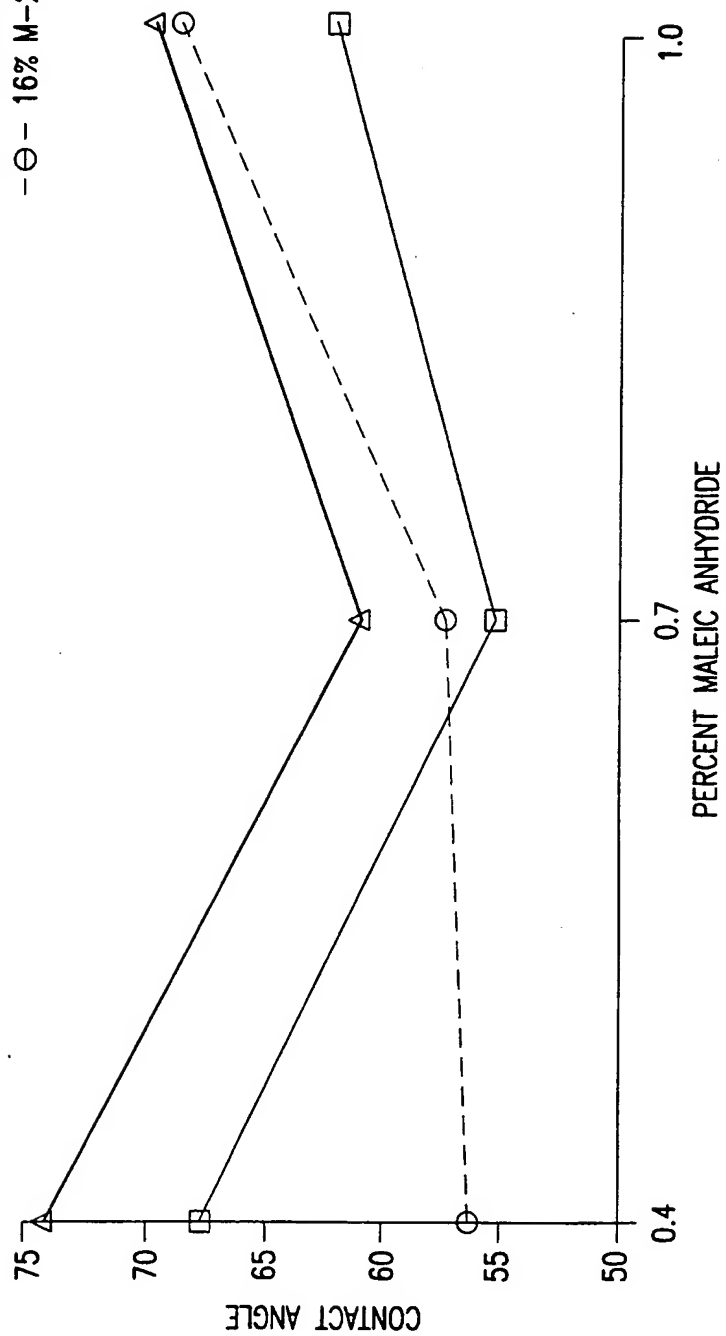


FIG.2

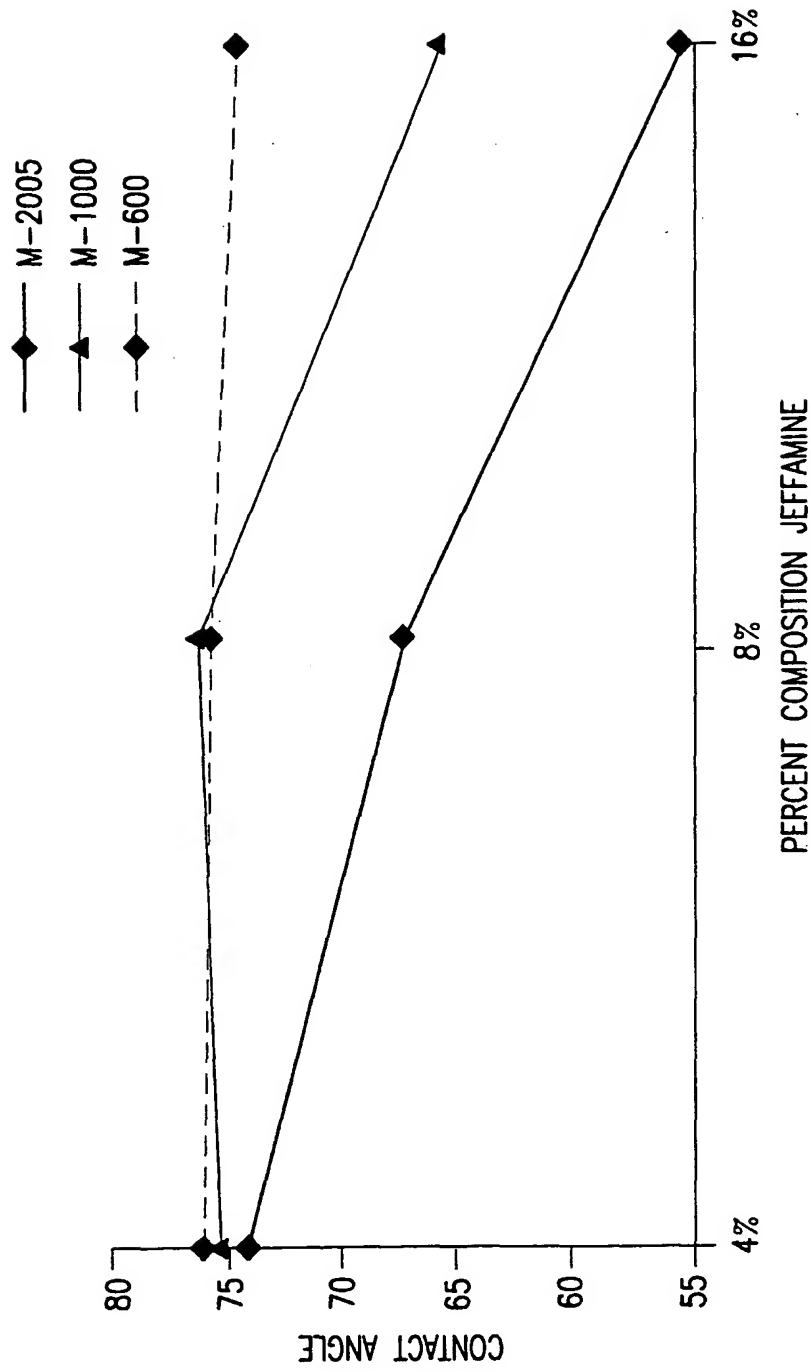


FIG.3

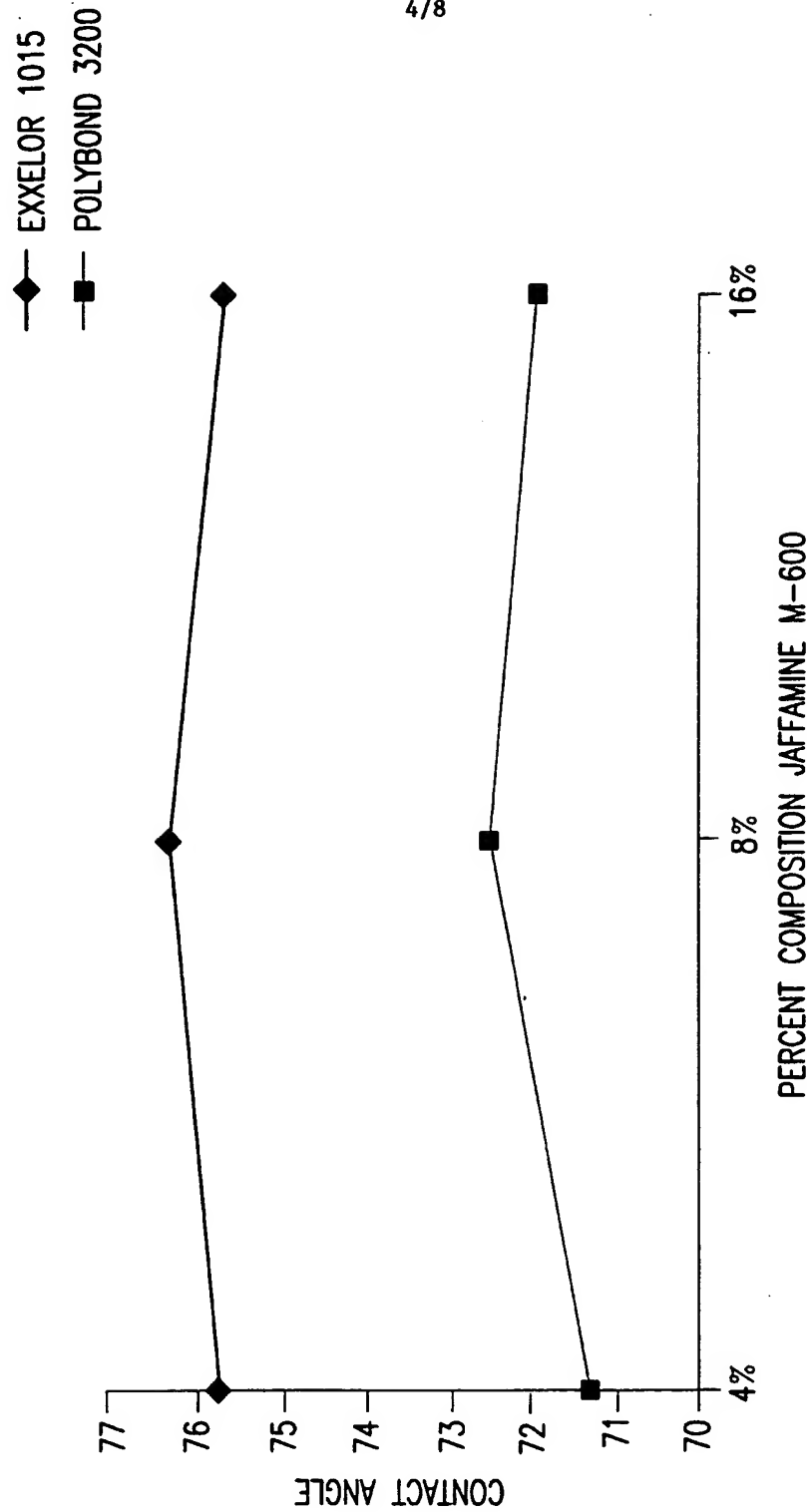


FIG.4

5/8

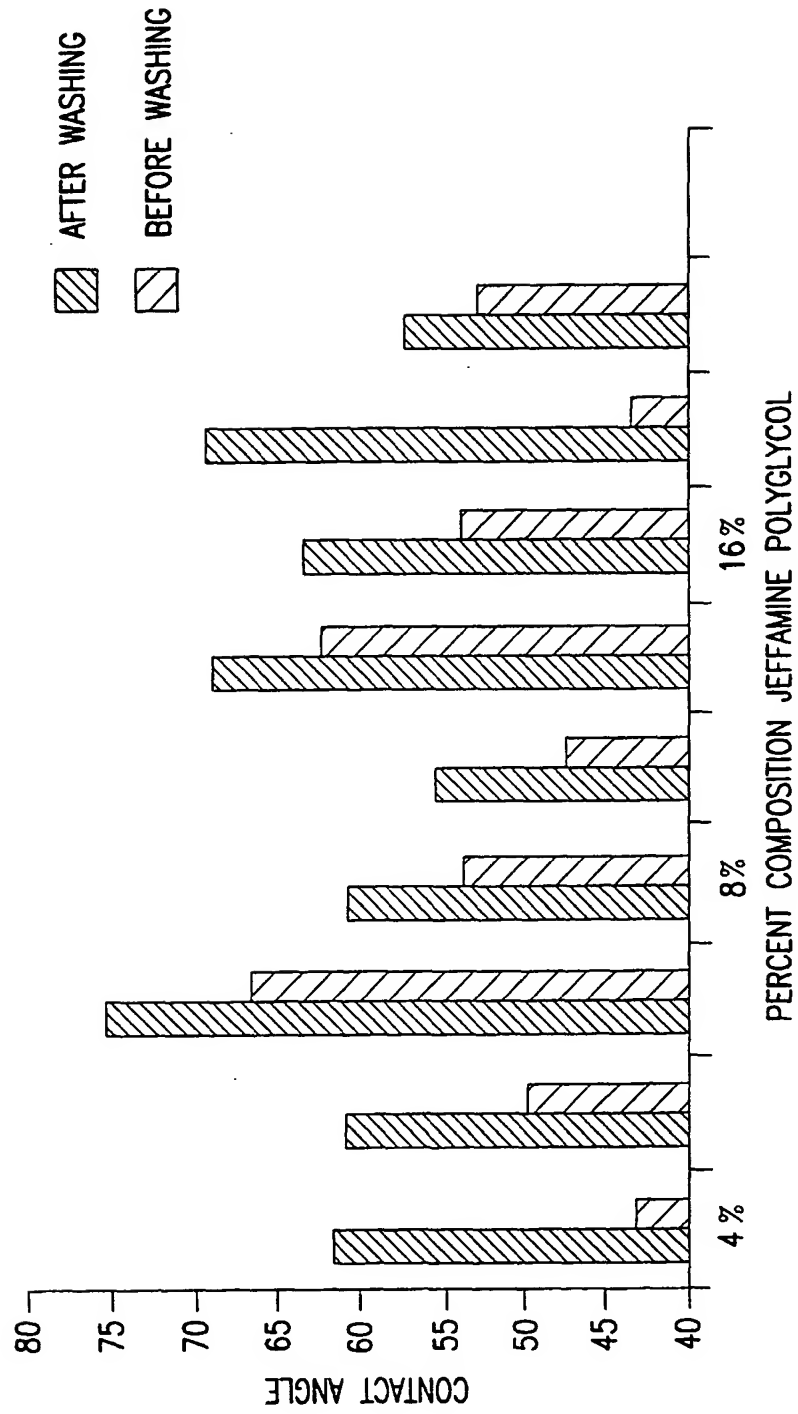


FIG. 5

6/8

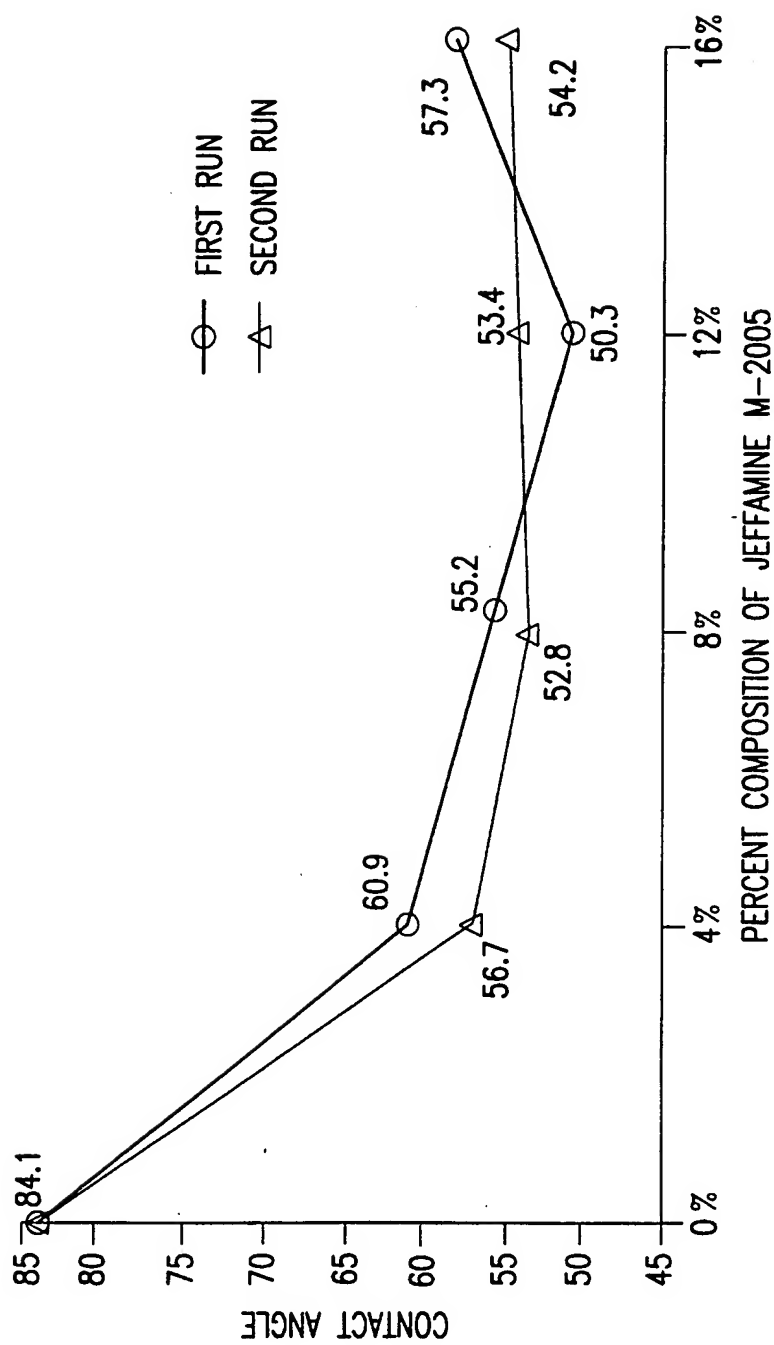


FIG.6

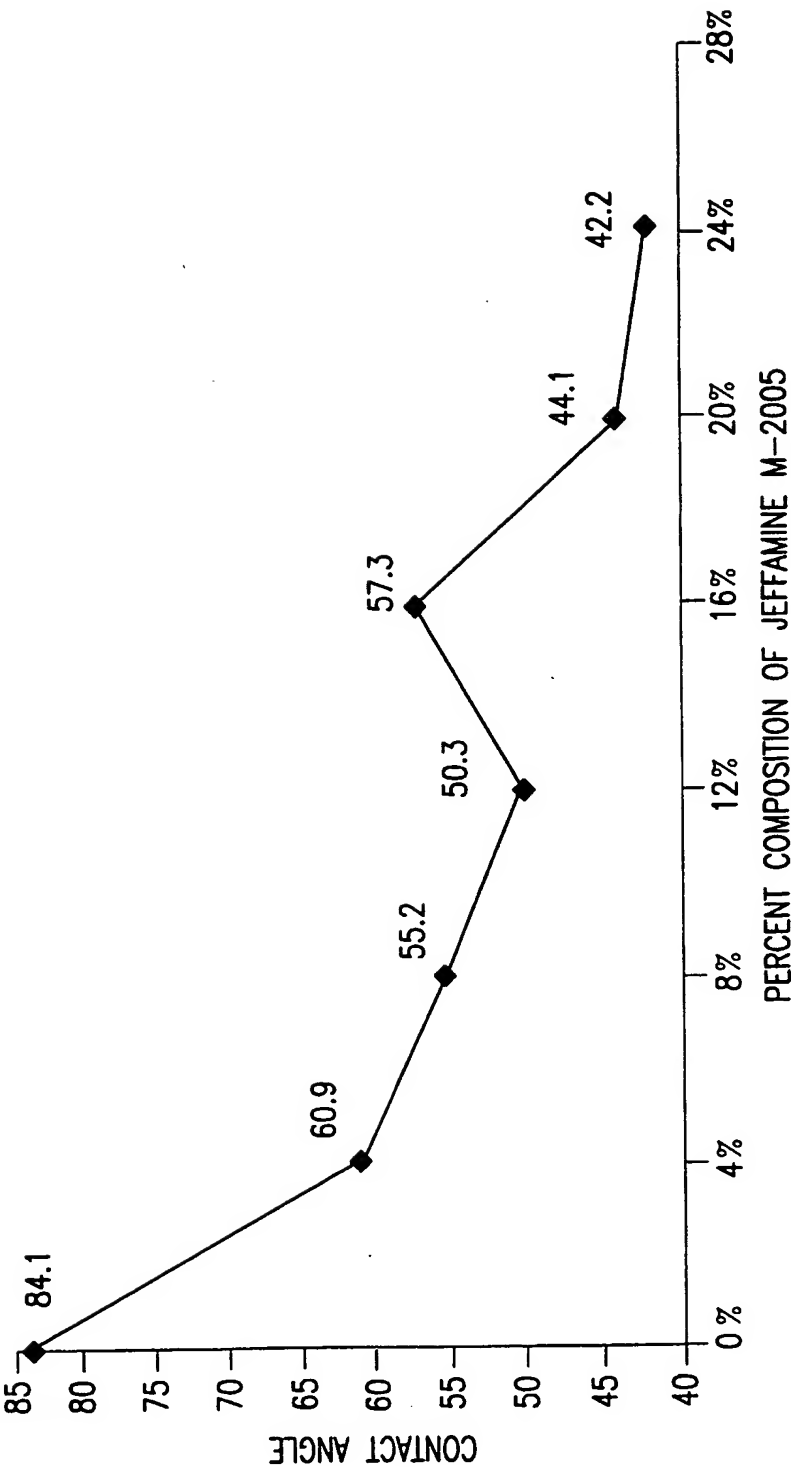


FIG.7

8/8

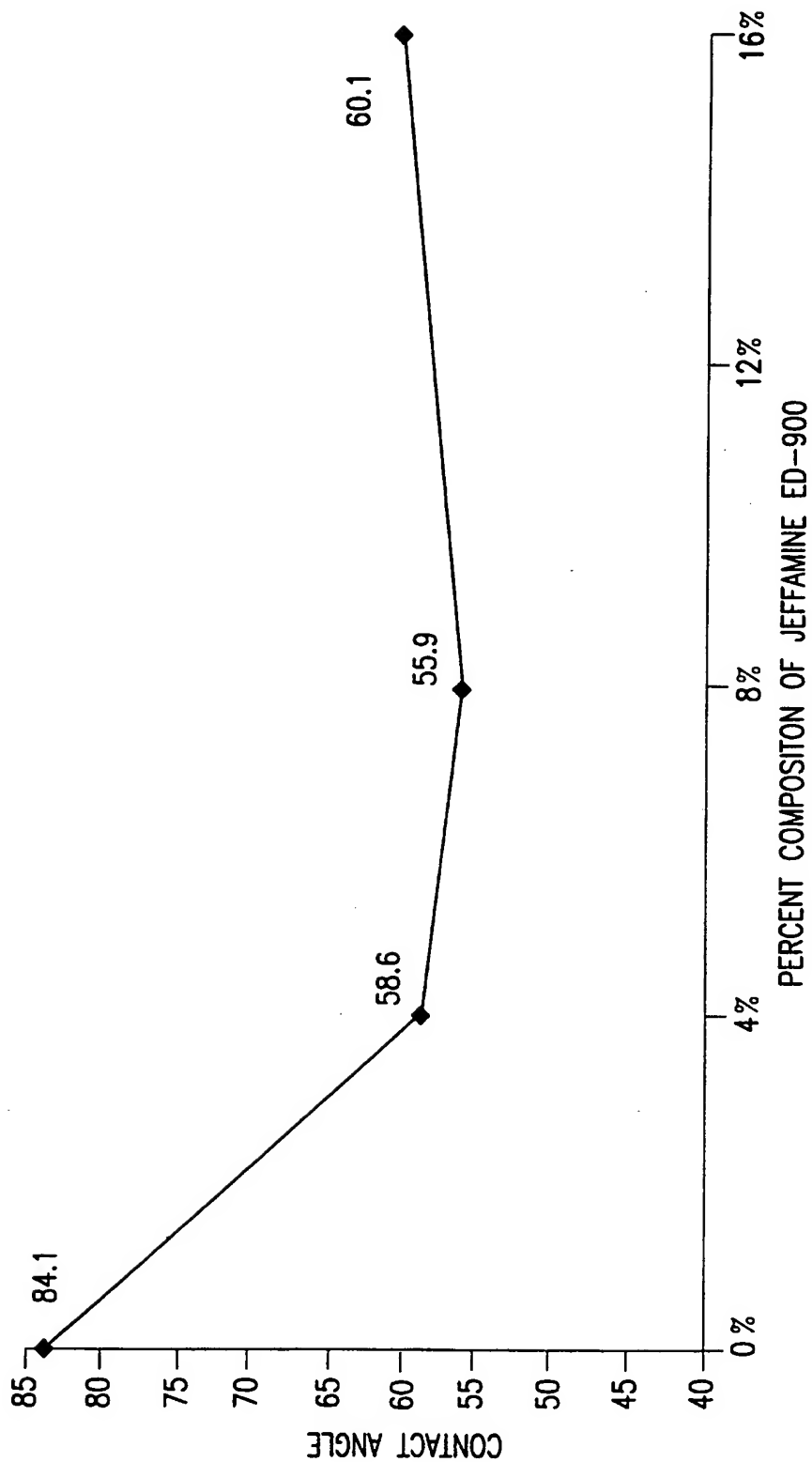


FIG. 8

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 99/18030

A. CLASSIFICATION OF SUBJECT MATTER IPC 7 D04H1/42 D01F6/30 A61L15/00		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC 7 D04H D01F A61L		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 114 379 A (MONTEDISON SPA) 1 August 1984 (1984-08-01)	1-14, 16-49
Y	the whole document -----	15
Y	EP 0 634 424 A (HUNTSMAN CORP) 18 January 1995 (1995-01-18) the whole document & US 5 721 315 A cited in the application -----	15
<div style="display: flex; justify-content: space-between;"> <input type="checkbox"/> Further documents are listed in the continuation of box C. <input checked="" type="checkbox"/> Patent family members are listed in annex. </div>		
<div style="display: flex;"> <div style="flex: 1;"> <p>* Special categories of cited documents :</p> <p>*A* document defining the general state of the art which is not considered to be of particular relevance</p> <p>*E* earlier document but published on or after the international filing date</p> <p>*L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>*O* document referring to an oral disclosure, use, exhibition or other means</p> <p>*P* document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="flex: 1;"> <p>*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>*&* document member of the same patent family</p> </div> </div>		
Date of the actual completion of the international search <div style="text-align: center; font-weight: bold;">29 November 1999</div>		Date of mailing of the international search report <div style="text-align: center; font-weight: bold;">13/12/1999</div>
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016		Authorized officer <div style="text-align: center; font-weight: bold;">Tarrida Torrell, J</div>

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 99/18030

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0114379 A	01-08-1984	IT 1155437 B	28-01-1987
		CA 1235832 A	26-04-1988
		DE 3378222 A	17-11-1988
		JP 5065622 B	20-09-1993
		JP 59130368 A	26-07-1984
		US 4636436 A	13-01-1987
EP 0634424 A	18-01-1995	CA 2127504 A	14-01-1995
		CN 1190413 A	12-08-1998
		DE 69403398 D	03-07-1997
		DE 69403398 T	25-09-1997
		JP 7145216 A	06-06-1995
		US 5783630 A	21-07-1998
		US 5721315 A	24-02-1998
		US 5959032 A	28-09-1999
		US 5942576 A	24-08-1999
		US 5965667 A	12-10-1999